POLYNUCLEAR AROMATIC HYDROCARBONS by EPA 610

Facility Name:_____VELAP ID_____

Assessor Name:_____Analyst Name:_____Inspection Date_____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst:_____ Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____					
Were sample bottles glass and either amber-colored or somehow protected from light?	5.1.1 9.1				
Were sample bottles and caps rinsed with acetone or methylene chloride and dried prior to use?	5.1.1				
If compositors were used, were sample containers protected from light and kept at 4°C?	5.1.2 9.1				
If compositors were used, was tubing not composed of Tygon and rinsed thoroughly with methanol followed by repeated rinsings with distilled water?	5.1.2 9.1				
Were sample containers not prerinsed with sample prior to filling?	9.1				
Were samples stored protected from light at 4°C from collection until extraction?	9.2				
Were samples tested for residual chlorine and, if necessary, dechlorinated with 80 mg sodium thiosulfate per liter of sample?	9.2				
Were samples extracted within 7 days of sampling, and were extracts analyzed within 40 days of extraction?	9.3				
Were interferents not observed in reagent water at the MDLs of anlytes of interest?	6.1				
Was $NaSO_4$ purified by muffling at 400°C for four hours?	6.5				
Was slica gel activated at 130°C for at least 16 hours prior to use?	6.6				
Were stock standards stored protected from light at 4°C and used for not longer than 6 months?	6.7.3				
Were at least three concentration levels used?	7.2.1 7.3.1				
Notes/Comments:					

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Was one of the standards near but above the MDL?	7.2.1 7.3.1				
When using an external calibration, was linearity through the origin assumed only if the standards had a less than 10% RSD?	7.2.2				
Were calibration factors or response factors used only if the standards had a less than 10% RSD?	7.2.2 7.3.2				
When using an internal calibration, was the method and matrix demonstrated to not interfere with the internal standard selected?	7.3				
Were the working calibration curves, response factors, and calibration factors verified each working day to be within ±15% without recalibration?	7.4				
Before using a cleanup procedures, were calibration standards subjected to it to demonstrate the absences of interferences?	7.5				
Were reagent water blanks analyzed to be free from interferences before sample analysis?	8.1.3				
Were spikes analyzed at a rate of a minimum of 10% of samples?(When analyzing less than 10 samples/month only 1 spike/month is required.)	8.1.4 8.3				
Were spike recoveries assessed against table 3 of the reference method?	8.3.3				
Were check standards analyzed at a rate of 10% of samples unless spikes demonstrated system control?	8.1.5				
Were check standards analyzed for analytes where spikes failed and found to fall within the criteria of table 3?	8.3.4				
Extraction					
Were sample containers marked for later determinations of sample volume prior to removal of samples?	10.1				
Were samples placed in separatory funnels?	10.1				
Were 60 mL portions of methylene chloride added to sample containers, the sample containers shaken for 30 seconds, and methylene chloride transferred to corresponding separatory funnel?	10.2				
Notes/Comments:					

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Were separatory funnels containing samples and methlyene chloride next shaken for two minutes?	10.2				
Were the phases in the separatory funnel allowed to separate for a minimum of 10 minutes?	10.2				
If emulsion layers between phases were more than one-third the volume of the solvent layer, were emulsion breaking techniques employed?	10.2				
Were methylene chloride layers collected from the separatory funnels into flasks?	10.2				
Were the steps of 10.2 repeated twice more with two more 60 mL volumes of methylene chloride?	10.3				
Were the three 60 mL volumes of methylene chloride extract collected together?	10.3				
Were the combined methylene chloride extracts passed through solvent-rinsed $NaSO_4$ drying columns and collected in a concentrator device?	10.5				
Were flasks and drying columns lastly rinsed with methylene chloride, and the rinsings added to the concentrator device?	10.5				
Were methylene chloride extracts concentrated to 1 mL?	10.6				
When extracts were stored for longer than two days, were they stored in Teflon-sealed screw-cap vials?	10.7				
Were original sample volumes determined to the nearest 5 mL using the markings on the sample containers?	10.8				
Cleanup and Separation (not necessary for clean sample matrices with no interferences)					
When cleanup procedures were used, was methylene chloride first exchanged to cyclohexane?	11.2				
Were columns composed of slurries of activated silica gel and methylene chloride underneath 1-2 cm anhydrous $NaSO_4$ prepared?	11.3.1				
Were columns preeluted with pentane, and the pentane discarded?	11.3.2				
Were cyclohexane extracts eluted through the columns followed by the elution of pentane before the $NaSO_4$ layer was exposed to air?	11.3.2				
Notes/Comments:					

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Were these pentane eluents also discarded?	11.3.2				
Were the columns eluted once more with methylene chloride, and this methylene chloride collected with sample extract?	11.3.3				
Were the sample extracts and methylene chloride layers concentrated to less than 10 mL?	11.3.3				
HPLC					
Were extraction solvents exchanged to acetonitrile, and acetonitrile concentrated to 1 mL?	12.1				
If internal standard was used, was internal standard injected into sample extract and the two mixed immediately before injection into the instrument?	12.4				
Was HPLC instrument allowed to equilibrate to gradient conditions between sample introductions?	12.5				
Were samples that exceeded the calibration range diluted and reanalyzed?	12.7				
GC					
Were the extracts concentrated to 1 mL?	13.2				
Were internal standards added to sample extracts and the two mixed immediately before introduction into the instrument?	13.5				
Were samples that exceeded the calibration range diluted and reanalyzed?	13.8				
Notes/Comments:					

Table 3—QC Acceptance Criteria—Method 610

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{X}$ (µg/L)	Range for P, P_s (%)
Acenaphthene	100	40.3	D-105.7	D-124
Acenaphthylene	100	45.1	22.1-112.1	D-139
Anthracene	100	28.7	11.2-112.3	D-126
Benzo(a)anthracene	10	4.0	3.1-11.6	12-135
Benzo(a)pyrene	10	4.0	0.2-11.0	D-128
Benzo(b)fluoranthene	10	3.1	1.8-13.8	6-150
Benzo(ghi)perylene	10	2.3	D-10.7	D-116
Benzo(k)fluoranthene	5	2.5	D-7.0	D-159
Chrysene	10	4.2	D-17.5	D-199
Dibenzo(a,h)anthracene	10	2.0	0.3-10.0	D-110
Fluoranthene	10	3.0	2.7-11.1	14-123
Fluorene	100	43.0	D-119	D-142
Indeno(1,2,3-cd)pyrene	10	3.0	1.2-10.0	D-116
Naphthalene	100	40.7	21.5-100.0	D-122
Phenanthrene	100	37.7	8.4-133.7	D-155
Pyrene	10	3.4	1.4-12.1	D-140

s = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, P_s = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

1.1 This method covers the determination of certain polynuclear aromatic hydrocarbons (PAH). The following parameters can be determined by this method:

Parameter	STORET No.	CAS No.
Acenaphthene	34205	83-32-9
Acenaphthylene	34200	208-96-8
Anthracene	34220	120-12-7
Benzo(a)anthracene	34526	56-55-3
Benzo(a)pyrene	34247	50-32-8
Benzo(b)fluoranthene	34230	205-99-2
Benzo(ghi)perylene	34521	191-24-2
Benzo(k)fluoranthene	34242	207-08-9
Chrysene	34320	218-01-9
Dibenzo(a,h)anthracene	34556	53-70-3
Fluoranthene	34376	206-44-0
Fluorene	34381	86-73-7
Indeno(1,2,3-cd)pyrene	34403	193-39-5
Naphthalene	34696	91-20-3
Phenanthrene	34461	85-01-8
Pyrene	34469	129-00-0